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JOHNS MANVILLE IP Department 10100 W Ute Avenue Littleton, Colorado 80127 (303) 978-2000

Case Docket No. 7395 Date: May 8, 2007

Mail Stop Appeals - Patents COMMISSIONER OF PATENTS PO Box 1450 Alexandria, VA 22313-1450

Re:

Application of: Hitch

Serial No.: 10/812,457 Filed: March 30, 2004

303-978-2323

Art Unit: 1771

Examiner: COLE, Elizabeth M.

For: NONWOVEN FIBER MATS WITH SMOOTH SURFACES AND METHOD

Transmitted herewith is/are the following document(s) related to the above-identified application:

- Notice of Appeal
- [X] Amended Appeal Brief
- Request for Oral Hearing

Please extend the time for filing the Notice of Appeal _____ () month to _

The fee has been calculated as shown below:

Notice of Appeal	\$500.00	
Appeal Brief	\$500.00	
Request for Oral Hearing	\$1000.00	
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1 month \$120.00, 2 m	nonths \$450.00, 3 months \$1020.00	
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Robert D. Touslee Registration No. 34,032 (303) 978-3927 Customer No. 29602

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In re Application of: Hitch

Art Unit: 1771

JOHNS MANVILLE

Serial No. 10/812,457

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For: NONWOVEN FIBER MATS WITH SMOOTH SURFACES AND METHOD

Commissioner of the Patents & Trademarks

Washington, D. C. 20231

Dear Sir:

In response to the Final Office Action mailed on March 30, 2006, the Advisory Action mailed May 10, 2006, Applicant has appealed the final rejection of the claims 1-8, 14 and 15, set forth in the Claims Appendix of this brief. This also is in response to the Notification of Non-Compliant Appeal Brief mailed April 19, 2007, the Third Amended Appeal Brief is submitted below.

THIRD AMENDED APPEAL BRIEF

Real Party in Interest:

The real party in interest is Johns Manville, assignee of the inventor, Hitch.

Related Appeals and Interferences

NONE

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Status of the Claims

303-978-2323

Claims 1-8, 14 and 15 were finally rejected under 35 USC 112, first and second paragraphs, and under 35 USC 102. Claims 3,4,7,8,14 and 15 stand finally rejected under 35 USC 103. A rejection of double patenting was overcome by the filing of a Terminal Disclaimer filed on May 3, 2006. Applicant appeals from the Final Rejection under 35 USC112, first and second paragraphs, 35 USC 102 and 35 USC 103. Claims 9-13 were cancelled.

Status of Amendments

After the Final Office Action mailed March 30, 2006, a Rule 1.116 amendment was filed. Though the Examiner had made new rejections in the Final Office action, the amendment was not entered and the Examiner maintained the Final Rejection.

Summary of the Claimed Invention:

Independent claim 1 involves nonwoven fibrous mats having a smooth surface for coating, page 1, lines 3-5 of the specification, and having a reduced amount of "stand-up" fibers, see page 2, lines 9-14, page 3, lines 5-9 and page 7, lines 6-8 and 15-19, the result of being comprised of a blend of at least two different lengths fibers, about 25 to about 50 wt. percent of the fibers being less than about 0.4 inch long, see page 3, lines 6-7 & 12 and page 7, lines 3-4 & 14, and about 75 to about 50 wt. percent of the fibers having a length of at least 0.45 inch long, see page 3, lines 11-2 and page 7, lines 2-3 & 13, all of the fibers being in the range of about 0.12 inch to about 0.6 inch long, see page 4, lines 16-17 and having a diameter in the range of about 9 -14 microns, see page 4, lines 15-16. These fibers line are bound together with about 10-25 wt. percent of a cured binder, see original claim 1. The combination of different fiber lengths in the ratio claimed produces an improved surface for painting or coating of the fibrous nonwoven mat, see Examples 2 and 3, particularly page 7, lines 6 & 16-19. Independent claim 5 is for a laminate comprising a layer of nonwoven fiber mat having a smooth surface for coating and a reduced amount of stand-up fibers, this mat being like the mat described in independent claim 1 with the exception being that all fibers have a length in the range of about 0.12-0.8 inch long instead of about 0.12-0.6 inch long, see page 3, lines 4-5, this mat being bonded to at least one layer of a different material, see page 3, lines 14-18.

The term "stand up fibers" refers to fibers that protrude from a surface of the mat various distances and make painting or coating of the surface difficult and cause visual defects in the painted or coated surface, see page 2, second full paragraph and the latter part of the third full paragraph. When many "stand up fibers" are present on the surface of the mat, typical of prior art mats, the surface of the mat requires a heavy coating to achieve a nice painted appearance, explained on page 2, lines 9-24, particularly important when the mat or coated mat is used as a facer on products such as gypsum wallboard, a typical application for the mats of the present invention. The mats of Examples 2 and 3 on page 7 of the specification, falling within the scope of claim 1, are compared with a conventional mat made in the same manner in Example 1 except that the fibers in the mat of Example 1 are conventionally all the same length. The mats of claimed invention have significantly fewer "stand up fibers", requiring less coating than prior art mats to produce a paintable surface, as pointed out in the last two sentences of Examples 2 and 3 on page 7.

Grounds of Rejection to be Reviewed on Appeal:

- 1. Claims 1-8, 14 and 15 stand finally rejected under 35 USC 112, first paragraph, as lacking descriptive support for, "about 25 to about 50 wt. percent fibers having a length of less than about 0.4 inch long and about 75 to about 50 wt. percent fibers having a length at least about 0.45 inch long."
- 2. Claims 1-8, 14 and 15 stand finally rejected under 35 USC 112, second paragraph as being vague and indefinite because of the phrase, "a reduced amount of stand up fibers."
- 3. Claims 1-8, 14 and 15 stand finally rejected under 35 USC 102(b) as being anticipated by the disclosure of Peng et al, U.S. Pat. Pub. No. 2003/0054714. The Final Office Action cites Peng et al as disclosing fibrous nonwoven mats allegedly having high tear strength comprising two different length fibers where from about 0 to about 100 wt. % of the fibers have an average length of from about 0.5 mm (0.0196 inch) to about 60 mm (2.362 inches) and about 0-100 wt. % of the fibers have average lengths of from about 10mm (0.394 in.) to about 150mm (5.91 in.), preferably comprising about 20 to about 80 wt. % fibers with an average length of about 10mm (0.394 in.) to about 45mm (1.772 in.) and about 20 to about 80 wt. % of fibers having an average length from about 30mm (1.181 in.) to about 80mm (3.15 in.), the fibers having a diameter of (1-100 microns) with (5-25

microns) preferred, and the mat having a binder content of 5-50 wt. %, the mat having an asphalt layers applied to its surface.

4. Claims 3-4, 7-8, and 14-15 stand finally rejected under 35 USC 103(a) as being obvious from the disclosure of Peng et al, described in # 3 above. The Final Rejection states that while Peng et al do not explicitly set forth the claimed ranges and proportions set forth in claims 2-3 (3-4?), 7-8 and 14-15, Peng et al does set forth using two different lengths of fibers to achieve high tear strengths and it would have been obvious for one of ordinary skill in this art to have selected the optimum lengths and proportions broadly disclosed by Peng et al through the process of routine experimentation to achieve optimum tear strength.

ARGUMENTS:

Ground #1 - Whether the specification provides descriptive support as required by 35 USC 112, first paragraph, for "about 25 to about 50 wt. percent fibers having a length of less than about 0.4 inch long and about 75 to about 50 wt. percent fibers having a length at least about 0.45 inch long."

The specification, which includes the original claims, does provide reasonable descriptive support for the limitations objected to for the following reasons:

1. The specification at page 3, in the Summary of the Invention, includes the following statement: .

"Preferably a major portion of the fiber is at least about 0.45 inch long and a minor portion of the fiber is shorter than about 0.4 inch."

It is well established that the claims are to be read in light of the specification and that the inventor is entitled to be his own lexicographer unless a term is used in a manner contrary to the ordinary meaning in the particular art most pertinent to the inventon. It is well established that the term "major portion" in a two component system, in this case the fiber system made up of two portions, the fibers of each portion having a different length, means "greater" than the other portion, e. g. "greater in quanity" according to Funk and Wagnall's NEW STANDARD OF THE ENGLISH LANGUAGE, 1943 edition. Claim 1 contains the term phrase, "blend of fibers of at least two different lengths". In a two fiber

length component system the major portion would be any quantity greater than about 50 wt. %, i.e. at least slightly larger than 50 wt. % such as 50.01 wt. % and the minor portion would be any quantity less than about 50 wt. % such as less than about 50 wt. % or less than 49.9999 wt. percent. This establishes the basis for the term "about 50 wt. percent" on each end of the range of the different length fiber portions.

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- 2. Also, original claim 13, a part of the specification, states: "The method of claim 9 wherein about half of the fiber is at least about 0.45 inch long and a remainder of the fiber is less than about 0.4 inch long."
- 3. Original claim 8 states: "The mat of claim 5 wherein the mat contains about 75 wt. percent fiber that is at least about 0.45 inch long and about 25 wt. percent fiber that is about 0.2 inch long."
- Example 3 illustrates using a blend of about 50 % 0.5 inch and about 50%
 0.2 inch fiber.
- 5. Example 2 illustrates using a blend of about 75 % .5 inch long fiber and about 25% fiber that was 0.2 inch long.

This disclosure clearly illustrates, and describes, to one of ordinary skill in the art that the "major portion" encompasses, at the very least, the range of about 50 wt. percent to about 75 wt. percent, and that the minor portion encompasses a range of about 25 wt. percent to about 50 wt. percent.

For these reasons Applicant believes the claims are in full compliance with the descriptive requirement of the first paragraph of 35 USC 112 and that the Examiner erred, and respectfully request the Board to reverse this rejection.

Ground #2 - Whether the term "a reduced amount of stand up fibers" renders the claim vague and indefinite under 35 USC 112, second paragraph.

It is well established that the claims are to be read in the light of the specification. The last two paragraphs of the Background in the Specification explains the shortcomings of the prior art mats regarding stand up fibers, and Example 1, a

representative prior art mat designed for coating, but shows requiring a heavy coating to cover up or reduce the "stand up" fibers. The second paragraph of the Summary of the Specification states that the invention "minimizes the amount of stand up fibers", thus it is clear that "reduced" means "reduced from prior art mats. Also, Examples 2 and 3 (inventive mats) show improved smoothness, reduction in stand up fibers, and requiring less coating material than the prior art mat of Example 1, a mat made for coating, and a mat containing only one length of fibers, 1/2 inch long 10-11 micron fibers and that requires a heavy coating due to the frequency and nature of the stand up fibers.

Further, the term "a reduced amount of stand up fibers" is a <u>property or characteristic</u> of the claimed mats, a result of following the compositional limitations. The mats with "a reduced amount of stand up fibers" is a result of compositional limitations of the claims and therefore is a <u>novel</u> property of the claimed mats, a property not easily defined precisely because both the frequency and the length of the stand up fibers is important to the desirability <u>and cost</u> of the <u>laminate containing the mat of the invention</u>. The frequency and the length of the stand up fibers above the surface of the mat is significant to the desirability of the mat for coating, see page 2, last 5 lines of the last full paragraph, that states,

"It is also known to use off-line coating to make mats having good hiding and painting properties since the <u>heavy coating</u> surrounds and holds down the 'stand up' fibers, but the <u>thick coating</u> required adds considerable cost to the product. If there were fewer 'stand up' fibers and if the ends of these fibers were closer to the surface of the mat, substantially less coating material would be required."

Typically, the most practical and most reliable test of the frequency and nature of the "stand up fibers" is best determined by what is required to produce a coated mat suitable for painting rather than a painstaking microscopic test that at best would look at a very small area of mat.

For these reasons applicant believes that the Examiner has erred in rejecting the claims as being indefinite under 35 USC 112, second paragraph, and respectfully requests the Board of Appeals to reverse this rejection.

Claims 14 and 15, dependent from claims 1 and 5 respectively, appear to be redundant and should depend from claims 2 and 6 respectively.

Ground # 3 - Whether the invention of all of the claims are anticipated under 35 USC 102(b) by the disclosure of Peng et al, U.S. Pat. Pub. No. 2003/0054714.

Claims 1-8 and 14-15 were rejected under 35 USC 102 as being anticipated by the disclosure of Peng et al, Pub. Pat. App. No. 2003/0054714. The Examiner urges that the statement, "mat comprises from about 0 to about 100 wt. percent of the fibers have an average length of from about 0.5 to about 60 mm and from about 0 to about 100 wt. percent of the fibers have an average length of about 10 to about 150 mm" anticipates the applicants claimed invention. Peng et al's preferred combination of 20-80% fibers about 10-45 mm long and 20-80% of the fibers about 30-80 mm is also so broad as to include 100% of the fibers that are 30-45 mm long, including a mat having 100 % of 1,18 1.25 Inch long fibers, and such is conventional for high tear mat. <u>These broad</u> statements of Peng et al about the fibers used in their invention, as the Examiner Is using it, is so broad as to include almost any nonwoven mat ever made and that ever will be made! Such broad statements do not anticipate every nonwoven mat made thereafter regardless of the fiber lengths, and particularly when the fiber lengths claimed are different than disclosed by the reference. For example, a broad, incredulous statement that "any chemical can be used as fuel" and citing gasoline as an example, cannot anticipate a later invention of using hydrogen peroxide as a fuel.

The Examiner's interpretation of this broad statement, i.e. that high tear mat can be made with 100 % short fibers such as less than 1/2 inch, without any evidence of correctness, is incredible, and not to be believed by one experienced in making nonwoven glass fiber mats, i.e. one of ordinary skill in the art. Allegations in any reference that are, because of the knowledge and experience of one of ordinary skill in the pertinent art, outrageous, are not to be taken as a legitimate teaching or reasonable suggestion that any embodiment falling within such a broad description meets the other descriptors of the invention. Note that this broad description of fiber lengths includes the prior art mat of present Example 1, shown by Examples 2 and 3 to be different in kind than the mats of the invention.

To be a legitimate teaching reference for the limitation or composition urged, the reference must not only enable the practice of that part of the invention, but also must first be credible in the mind of one of ordinary skill in the art. This reference fails in both with respect to the broad statement relied on by the Examiner because the <u>limitations</u> are so broad as to be obviously unecessary. These limitations include 0-100 % of

the fibers to be .5 mm (about 1/50th of an inch) to about 150 mm (about 6 inches). This ridiculously broad statement is <u>so broad as to not require a blend of two fiber lengths.</u>

Further, the shortest commercially available glass fibers are no shorter than about 0.067 (1/16th) inch long and the longest chopped fibers commercially available are shorter than 3 inches (about 76 mm) long. Therefore this broad statement of using fibers of about 1/50th of an inch long to about 6 inches long, the statement that the Examiner is relying on, is <u>incredulous to one of ordinary skill in the art</u>. In wet laid processes that Peng et all prefers, it is not known how to disperse and form a suitable mat from chopped fibers longer than about 3 inches, and practically, not longer than about 2 inches long. Those skilled in the art would not believe that part of Peng et al's broad statement that is being urged to encompass these limitations of the present invention.

Such a broad statement does not even warrant testing because those skilled in the art, knowing the effect of fiber length on tear strength of the mat, and the roofing product containing the mat, such as shingles, know that mats containing mixtures containing any significant amount of glass fibers of less than 3/4 -1/2 inch long cannot have tear strengths better than prior art roofing mats and ditto for roofing products, such as shingles. Note that Peng et al do not reasonably show that the fiber lengths of this broad range produce the high tear strengths in the mat and roofing product, the objective of the invention. Instead, only fibers having lengths of 30-40 mm (1.18 - 1.6 inches) are shown to meet the objectives of high or higher tear strength. These long fiber lengths are conventional in high tear strength mats. Therefore, those of ordinary skill in the art would conclude that the presence of fibers less than .8 or particularly less than .6 inch long (current spec., first paragraph of Detailed Description of the Invention) do not produce higher tear strength mat. Reading applicant's claims in light of the specification shows that the term "at least .45 inch long" is not unlimited on the maximum length, but instead is limited by the following disclosure taken from the first paragraph of Detailed Description of the Invention, which reads:

"A slurry of fibers is made by adding the fibers, having an average fiber diameter from about 9 to about 14 microns and lengths, preferably a blend of two lengths, of from about 0.12 to about 0.8 inch, preferably 0.2 to 0.6 inch, long, -----" (emphasis added)

The higher tear strength claimed by Peng et al is due to the presence of polysiloxane primarily and because the use of fibers as long as 30- 40 mm fibers is well known. This is acknowledged by Peng et al in the paragraph 0035. Peng et al's disclosure that higher basis weight in the mat produces higher tear strength is also well known. Therefore, Peng et al would not change the mindset of one of ordinary skill in the art that short glass fibers having lengths of less than about 1 inch long such as 1/2 inch long of shorter do not produce high tear strength in nonwoven mat. This is evidenced by Examples 1-7 of Peng et al and also all the Examples of a different inventor working for a different company, see US 5,518,586 discussed in Peng et al, see paragraph [0006]. In 5,515,586 the invention is a method of making a nonwoven glass fiber mat having high tear strength by modifying a urea formaldehyde resin binder in a particular way, the mat being particularly useful in shingles. Note that while a range of fiber lengths of 1/4 inch to 3 inches are said to be useful, the fiber length used in all of the examples is 1 inch long, col. 5, lines 60-62 col. 6, lines 14-15 and 52-53 and col. 7, lines 15-16 and 58-59.

A broad statement, certainly a ridiculously broad statement like the one being relied on by the Examiner, does not teach or reasonably suggest, even prima facie, the much more narrow limitation, such as fiber lengths of less than 1/2 inch, that produces results not taught or reasonably suggested by the reference - here a smoother surface and fewer "stand up fibers". For example, the broad teaching that any organic resin will work cannot be a reasonable teaching, in the sense of 35 USC 102, that the use of a specific family of resins will produce results substantially different than taught by the reference. The Field of the Invention and the first sentence of both the Summary of the Invention and the Detailed Description of the Peng Invention states that the mats of the invention have "improved tear strength" that results in roofing products requiring improved tear strength". All of the examples shown by Peng et al used fibers. that were all at least 30 mm, i.e. 1.18 inches long! Peng et al does not teach how to make a roofing product having a high tear strength or any other product having improved tear compared to prior art products using a blend of fiber lengths like those described in the rejected claims. Further, the Peng et al reference is silent about the surface characteristics their mats.

If, on the other hand, Peng et al, by the use of this broad statement, is merely meaning that his invention, the application of polydimethylsiloxane to a wet web of glass fibers and polymer binders, is applicable to an extremely broad range of fiber lengths or

an extremely broad range of mixed fiber lengths, which might be the case, then such statements cannot be reasonably interpreted to mean what the Examiner is urging it means, i.e. that Peng et al teaches making mats for roofing and building products having improved tear strengths, compared to prior art products, using any combination of fiber lengths and percentages within his broad statement(s).

Finally, for a reference to anticipate an invention it must teach every element of the claim(s). Peng et al do not teach every element of the rejected claims.

One example, Peng et al does <u>not</u> teach using 9-14 micron diameter fibers, the mats described in claims 3 and 4, or the laminates of claims 5-8, 14 and 15. For example, teaching using fibers within the range of 1-100 microns in diameter <u>does not teach</u> using fibers in a range of 9-14 microns and teaching using a combination of 0-100 wt. percent of fibers of one length and 100-0 wt. percent of fibers of a second length does not teach a mat of comprising fibers in which 25 wt. percent of the fibers are one length and 75 wt. percent of the fibers are a different length.

Another example, claims 3, 5 and 14-15 describe a mat in which about half the fiber is at least 0.45 inch long and the remainder of the fiber is less than 0.4 inch long. Peng et al does not teach or <u>reasonably suggest such a combination</u>.

A still further example, claims 4 and 6 describe a mat in which about 75 wt. percent of the fiber is at least about 0.45 inch long and about 25 wt. percent of the fiber is about 0.2 inch long.

For these reasoms applicant believes that the Examiner has erred in rejecting claims 1-8 and 14-15 under 35 USC 102(b) and respectfully requests the Board to reverse this rejection.

Ground #4 - Whether the invention of claims 3-4, 7-8, and 14-15 is made obvious under 35 USC 103(a) by the disclosure of Peng et al, described in Issue 3 above.

The Examiner urges that the very broad fiber length combinations disclosed in Peng et al makes it obvious to one of ordinary skill in the art to optimize these combinations to achieve maximum tear strength and that this makes the presently claimed mats and laminates, containing fibers about 0.2 inch long and fibers about 0.45-0.8 inches long, of claims 3-4, 7-8 and 14-15 obvious under 35 USC 103. Although tear

strength data for the mats of the claimed invention are not presented in the specification because tear strength was not important, the mats of the invention do not have high tear strengths, but instead have smooth surfaces and a substantial reduction in "stand up fibers" compared to prior art mats, a completely different objective and result than the mats of Peng et al.

More importantly, this assertion is contrary to the evidence present in Peng et al and at least one patent discussed in Peng et al. As pointed out above in the arguments in Issue # 3, both Peng et al and the inventor Mirous in US Pat. No. 5,515,586, teach methods of making a nonwoven glass fiber mat having high tear strength for use in shingles. In setting forth the best mode, both references use fibers that are at least one inch long (Note that the fiber length used in all of the examples in '586 is 1 inch long, see col. 5, lines 60-62 col. 6, lines 14-15 and 52-53 and col. 7, lines 15-16 and 58-59). The fiber lengths used By Peng et al in the best mode shown in the Examples are at least 30 mm, 1.16 inches, long and up to 40 mm, 1.57 inches long. Therefore, the Peng et al disclosure leads those of ordinary skill in the art away from the claimed invention, mats along or in laminates containing fibers from 0.12 Inch long to 0.8 inches long. When a reference leads those skilled in the art away from the invention, it cannot be maintained that the reference makes the Invention obvious under 35 USC 103.

Respectfully submitted,

Date: May 8, 2007

P.O. Box 625005 Littleton, CO 80162 Robert D. Touslee Registration No. 34,032

Tel. (303-978-3927)

Appendix - Claims

List of claims involved in the appeal:

- 1. (Previously presented) A nonwoven fibrous mat having a smooth surface for coating, the smooth surface having a reduced amount of "stand up fibers" comprising a blend of fibers of at least two different lengths falling within the range of about 0.12 inch and about 0.6 inch bound together with a cured binder, the binder content of the mat being in the range of about 10-25 weight percent of the finished mat, the fibers having an average fiber diameter in the range of about 9 and about 14 microns and about 25 to about 50 wt. percent fibers having a length of less than about 0.4 inch long and about 75 to about 50 wt. percent fibers having a length at least about 0.45 inch long.
- 2. (Original) The mat of claim 1 wherein the binder is selected from a group consisting of an acrylic, a polyvinyl alcohol, a hydroxyl ethyl cellulose, a carboxyl methyl cellulose, a cellulose gums, a polyvinyl pyrilidone, polyvinyl acetate, urea formaldehyde, melamine formaldehyde, with or without a crosslinking agent, with or without one or more plasticizers, and mixtures thereof.
- 3. (Original) The mat of claim 1 wherein about half of the fiber is at least about 0.45 inch long and a remainder of the fiber is less than about 0.4 inch long.
- 4. (Previously presented) The mat of clalm 1 wherein the mat contains about 75 wt. percent fiber that is at least about 0.45 Inch long and about 25 wt. percent fiber that is about 0.2 inch long.
- 5. (Previously presented) A laminate comprising a layer of nonwoven fiber mat having a smooth surface for coating, the smooth surface having a reduced amount of "stand up fibers" and comprising a blend of fibers of at least two different lengths falling within the range of about 0.12 inch and about 0.8 inch bound together with a cured binder, the binder content of the mat being in the range of about 10-25 weight percent of the finished mat, the fibers having an average fiber diameter in the range of about 9 and about 14 microns, and about 25 to about 50 wt. percent fibers having a length of less than about 0.4 inch long and about 75 to about 50 wt. percent fibers having a length at least about 0.45 inch long, a surface of said mat being bonded to at least one layer of different material.

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- 6. (Original) The laminate of claim 5 wherein the binder is selected from a group consisting of an acrylic, a polyvinyl alcohol, a hydroxyl ethyl cellulose, a carboxyl methyl cellulose, a cellulose gums, a polyvinyl pyrilidone, polyvinyl acetate, urea formaldehyde, melamine formaldehyde, with or without a crosslinking agent, with or without one or more plasticizers, and mixtures thereof.
- 7. (Original) The laminate of claim 5 wherein about half of the fiber is at least about 0.45 inch long and a remainder of the fiber is less than about 0.4 inch long.
- 8. (Original) The mat of claim 5 wherein the mat contains about 75 wt. percent fiber that is at least about 0.45 inch long and about 25 wt. percent fiber that is about 0.2 inch long.
- 9-13 (Cancelled)
- 14. (Original) The mat of claim 1 wherein about half of the fiber is at least about 0.45 inch long and a remainder of the fiber is less than about 0.4 inch long.
- 15. (Original) The laminate of claim 5 wherein about half of the fiber is at least about 0.45 Inch long and a remainder of the fiber is less than about 0.4 inch long.

EVIDENCE (ADDITIONAL) APPENDIX

US Pat. No. 5,518,586 - Mirous (Copy Attached)

I DEBIAN KERBANA BUJARAN ETABU BUNKA MAKAT KUMAN KARIN MAKAT FIRIN BUJAN PERBANJAN KUMAN KARIN PERBA US005518586A

United States Patent [19]

303-978-2323

Mirous

[45] Date of Patent:

5,518,586

[54] METHOD OF MAKING A HIGH TEAR STRENGTH GLASS MAT

- [75] Inventor: George E. Mirous, Tacoma, Wash.
- [73] Assignce: Georgia-Pacific Resins, Inc., Atlanta, Ga.
- [21] Appl. No.: 450,151
- [22] Filod: May 26, 1995

Related U.S. Application Data

- [62] Division of Scr. No. 123,094, Sep. 20, 1993, Pat. No. 5,445,878.
- [51] Int. CL* [52] U.S. Cl. 162/156; 162/158; 162/167;
- 162/186; 156/62.2 [58] Field of Scarch 162/156, 167, 162/158, 166, 186, 184; 156/62.2; 264/109

[56]

References Cited

U.S. PATENT DOCUMENTS:

2,906,660 9/1959 Hungerford et al. . 3,012,929 12/1961 Jackson . 3,050,427 8/1962 Slayter et al. . 3,103,461 9/1963 Smith et al. .

[11] Patent Number:

May 21, 1996

1/1966 Waggoner . 3,228,825 3,760,458 9/1973 Pitt . 3,766,003 10/1973 Schmiller et al. . 3,838,995 10/1974 Smith . 3,905,067 9/1975 Keib et al. 4.178,203 12/1979 Chakrabarti . 4,210,462 4,210,562 7/1980 McCombs. 7/1980 McCombs 4,258,098 3/1981 Bondoc et al . 4,430,158 2/1984 Jackey et al. . 7/1984 Hsu et al. . 4,457,785 4,536,446 8/1985 Hsn et al. . 4,681,658 7/1987 Hsu et al. . 4,683,165 7/1987 Linderman et al., 4,917,764 4/1990 Lalwani et al. . 3/1993 Lindemann et al. 5,190,997 5,219,656 6/1993 Klen et al. . Primary Examiner-Michael W. Ball

Assistant Examiner-Sam Chuan Yao Attorney, Agent, or Firm-Banner & Allegretti, Ltd.

ABSTRACT.

A mea-formaldehyde resin modified with a water-insoluble anionic phosphate ester is used as binder in the preparation of glass fiber mass using a hydroxyethyl cellulose white water system. High toar strength glass fiber mats can be produced in a hydroxycthyl cellulose white water system using such a hinder.

2 Claims, No Drawings

1

METHOD OF MAKING A HIGH TEAR STRENGTH GLASS MAT

This application is a division of application Ser. No. 08/123,094, filed Sep. 20, 1993, now U.S. Pat. No. 5,445, 5878

BACKGROUND OF THE INVENTION

1. Field of the Invention

The invention relates to a modified urea-formalde-hyde resin, to glass fiber mats using the modified urea-formalde-hyde resin as binder, and a process of preparing the mats. In particular, the invention relates to a urea-formaldehyde resin modified with a water-insoluble anionic phosphate ester 15 which is useful in the preparation of glass fiber mats formed using a hydroxyethyl cellulose-containing "white water" glass slurry. The glass fiber mats of the invention exhibit high tear strength, a property which is desirable for use in 20 roofing products, such as asphalt shingles.

2. Background of the Invention

Glass fiber mats are finding increasing application in the building materials industry, as for example, in asphalt roofing shingles, replacing similar sheets traditionally made of wood or cellulose fibers.

Glass fiber mats usually are made commercially by a wet-laid process, which is carried out on modified paper or asbestos making machinery. Descriptions of the wet-laid 30 process may be found in a number of U.S. patents, including U.S. Pat. Nos. 2,906,660, 3,012,929, 3,050,427, 3,103,461, 3,228,825, 3,760,458, 3,766,003, 3,838,995 and 3,905,067. In general, the known wet-laid process for making glass fiber mats comprises first forming an aqueous slurry of short-length glass fibers (referred to in the art as "white water") under agitation in a mixing tank, then feeding the slurry through a moving screen on which the fibers enmesh themselves into a fleshly prepared wet glass fiber mat, while water is separated therefrom.

Unlike natural fibers such as cellulose or asbestos, glass fibers do not disperse well in water. In an attempt to overcome this problem, it has been the practice in the industry to provide suspending aids for the glass fibers. Such suspending aids usually are materials which increase the viscosity of the medium so that the fibers can suspend themselves in the medium. Suitable dispersants conventionally employed in the art include polyacrylamide, hydroxysethyl cellulose, ethoxylated applies and amine oxides.

Other additives such as surfactants, hubricants and defoamers have conventionally been added to the white water. Such agents, for example, aid in the wettability and dispersion of the glass fibers and countibute to the strength of the wet glass fiber mat. U.S. Par. No. 4,178,203 is directed to a method for improving the wet tensile strength of freshly prepared glass fiber mats so that they may be conveniently handled and transferred for further processing (e.g., applying binders and drying) to form the finished glass fiber mat product. In the disclosed process, anionic surfactants are added to the white water glass shury.

In the manufacture of glass met, a high degree of flexibility and tear strength is desired in addition to the primary dry tensile and hot wet tensile properties. A binder material 2

is therefore used to hold the glass fiber mat together. The binder material is impregnated directly into the fibrous mat and set or caused to provide the desired integrity. The most widely used binder is urea-formaldehyde resin because it is inexpensive.

While urea-formaldehyde resins are commonly used to bond the glass fibers together to provide the strength properties of the glass mat, some urea-formaldehydo resin binders are too brittle to form glass mats useful in roofing shingles. Typically, the tensile strengths of mass bound with urea-formaldchyde deteriorate appreciably when the mats are subjected to wet conditions, such as the conditions normally encountered by roofing products. Tear strengths higher than those typically provided by urea-formaldchyde resins have been obtained by modifying the resin with cross-linkers and various catalyst systems or by fortifying the resin with a large amount of latex polymer, usually a polyvinyl acetate, vinyl acrylic or styrene-butadiene. Latex provides increased hot wet tensile strongth and tear strength. The use of styrene-butadiene modified urea-formaldehyde resins as a binder for glass fiber mats is disclosed, for example, in U.S. Pat. Nos. 4,258,098 and 4,917,764.

U.S. Pat. No. 4,430,158 is directed to an improved binder composition for glass mats. The binder composition consists essentially of a urea-formaldehyde resin and a highly water soluble anionic surfactant that wets the surfaces of the glass fibers. Suitable surfactants have hydrophobic segments containing from 8 to 30 carbon atoms and anionic acgments. Suitable anionic moieties include carboxy, sulfate ester, phosphate ester, sulfonic acid, and phosphoric acid groups. The surfactant also may contain a polyalkyleneoxy chain having up to 10 alkyleneoxy units. Glass mats produced from an amine oxide white water system and bound with the surfactant-containing resin, are described as retaining up to 79 percent of their dry tensile strength when subjected to severe wet conditions. No increase in tear strength is obtained by use of the urea-formaldehyde surfactant-containing resin. Cationic surfactants, non-ionic surfactants, and anionic surfactants which do not possess the required water solubility and ability to wet the sized glass fibers, are said to provide unsultable mats which can retain a much smaller fraction of their dry tensile strength.

When the class fibers are dispersed in white water containing a polyacrylamide viscosity modifier, high tear mat strengths have been achieved with latex fortification of urea-formaldchyde resins. However, when a hydroxyethyl cellulose viscosity modifier is used in the white water, the desired high tear strength properties are not achieved with latex fortification. As such, a need in the art exists for providing a modified urea-formaldchyde resin which can be used in a hydroxyethyl cellulose white water system.

SUMMARY OF THE INVENTION

The invention is directed to a modified urea-formaldehyde resin. The invention also is directed to a process for preparing glass fiber mats, and to glass fiber mats produced by the method. The mats are useful in, for example, the manufacture of roofing shingles.

This invention is based on the discovery that by adding a water-insoluble anionic phosphate ester to a urea-formalde-

and the like.

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hyde resin, high tear strength products can be prepared from mats formed using hydroxyethyl cellulose-containing white water.

In manufacturing glass fiber mats in accordance with the invention, glass fibers are slurried into an aqueous medium containing hydroxyethyl cellulose. This white water, i.e., the hydroxyethyl cellulose-containing slurry of glass fibers in water, then is downtered on a foraminated surface to form a mat. The modified binder of the invention then is applied to the mat before it passes through a drying oven where the mat is dried and incorporated binder resin is cured. Glass fiber mats produced in accordance with the invention exhibit good dry and hot wet tensile strength and superior high tear strength.

One object of the invention is to provide a binder composition for use in making glass fiber mats comprising a urea-formaldehyde resin and a water-insoluble amonic phosphate ester.

Another object of the invention is to provide glass fiber mats comprising a wea-formaldehyde resin and a waterinsoluble anionic phosphate ester.

Yet another object of the invention is to provide glass fiber 2s mats prepared by dispersing glass fibers in an aqueous medium containing hydroxyethyl cellulose to form a slurry, passing the slurry through a mat forming screen to form a wet glass fiber mat, applying a binder comprising a ureaformaldehyde resin and a water-insoluble anionic phosphate ester to said wet glass fiber mat, and curing the binder.

DETAILED DESCRIPTION OF THE INVENTION

Urea-formaldehyde resins have been modified with crosslinkers and various camlyst systems or fortified with large amounts of latex to achieve high glass mat tear strengths in mats processed in polyacrylamide-containing white water. 40 However, such modified and fortified resins have no effect in a hydroxyethyl cellulose-containing white water system. It has now been discovered that the modification of ureaformaldehyde resin with a water-insoluble anionic phosphate ester as a binder for glass mat obtained from a hydroxyethyl cellulose-containing white water system not only provides higher tear strength without a loss in dry or het wet tensile properties, but also does not require latex fortification. This not only eliminates handling and clean up 50 problems associated with latexes, but is also significantly lower in cost,

The process of forming a glass fiber mat in accordance with the invention begins with chopped bundles of glass fibers of suitable length and diameter. While reference is made using chopped bundles of glass fibers, other forms of glass fibers such as continuous strands may also-be used. Generally, fibers having a length of about ¼ inch to 3 inches and a diameter of about 3 to 20 microns are used. Each 60 bundle may contain from about 20 to 300, or more, of such fibers.

The glass fiber bundles are added to the dispersant medium to form an aqueous slurry, know in the art as "white 65 water." The white water typically contains about 0.5% glass. The dispersant used in the practice of the invention contains

hydroxycthyl cellulose. The amount of hydroxycthyl cellulose used should be effective to provide the viscosity needed to suspend the glass particles in the white water. The viscosity is generally in the range of 5 to 20 cps, preferably 12 to 14 cps. An amount of from about 0.1 to about 0.5% solid hydroxyethyl cellulose in the water should be sufficient. The fiber/white water mixture generally is at a temperature of 65° to 95° F. to obtain preferred viscosity. The fiber slurry then is seltated to form a workable uniform dispersion of glass fiber having a suitable consistency. The dispersant may contain other conventional additives known in the art. These include surfaciants, lubricants, defoamers

The fiber/white water dispersion then is passed to a mat-forming machine containing a mat forming screen. On mute to the screen, the dispersion usually is diluted with water to a lower fiber concentration. The fibers are collected at the screen in the form of a wet fiber mat and the excess water is removed by gravity or, more preferably, by vacuum in a conventional manner.

The binder composition of the invention then is applied to the gravity- or vacuum-assisted dewatered wet glass mat. Application of the binder composition may be accomplished by any conventional means, such as by soaking the mat in an excess of binder solution, or by coating the mat surface by means of a binder applicator.

The urea-formaldehyde resin used as binder in the invention is a urea-formaldehyde resin modified with an anionic phosphate ester. The anionic phosphate esters useful in the invention are water insoluble. Particularly preferred anionic phosphate esters are unneutralized water insoluble phosphate esters, such as the type exemplified by ZELEC UN® available from Du Pont. ZELEC UN® is an unneutralized, water-insoluble anionic phosphate ester with a high molecular weight a C₃ of C₁₆ fatty alcohol backbone. Stated another way, ZELEC UN® is an unneutralized water-insoluble, anionic phosphate C₃ of C₁₆ alkyl ester of phosphoric acid and a fatty alcohol. A urea-formaldehyde resin modified with ZELEC UN® has been found to be particularly advantageous in the preparation of glass fiber mats having high tear strength from hydroxyethyl cellulose white water.

Methods of preparing urea-formalchyde resins which may be used to prepare the binder composition of the invention are known to those skilled in the art. Many urea-formaldehyde resins which may be used in the practice of the invention are commercially available. Urea-formaldehyde resins such as the types sold by Georgia Pacific Corp. for glass mar application and those sold by Borden Chemical Co., may be used. These resins generally are modified with methylol groups which upon curing form methylene or ether linkages. Such methylols may include N,N'-dimethylol, dihydroxymethylolethylene; N,N' bis-(methoxymethyl), N,N'-dimethylolpropylene; 5,5-dimethyl-N,N' dimethylolpropylene; and the like.

The binder composition is prepared by rapidly dispersing the anionic phosphate ester into the urea-formaldehyde resin having a pH of 7.5 to 8.5. If needed pH of the resin is adjusted to 7.5 to 8.5 with caustic. The amount of phosphate ester is about 0.1 to about 5.0%, preferably about 0.5% of the blader composition.

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Urea-formaldehyde resins useful in the practice of the invention generally contain 45 to 65%, preferably, 50 to 60% non-volatiles, have a viscosity of 50 to 500 cps, preferably 150 to 300 cps, a pH of 7.0 to 9.0, preferably 7.5 to 8.5, a free formaldehyde level of 0.0 to 3.0%, preferably 0.1 to 0.5%, a mole ratio of formaldehyde to urea of 1.1:1 to 3.5:1, preferably 1.8:1 to 2.1:1, and a water dilutability of 1:1 to 100:1, preferably 10:1 to 50:1.

Whereas high tear strength mats can be prepared using 10 latex-fortified binders when the white water additive is polyacrylamide, high strength mats have not heretofore been prepared using hydroxyethyl cellulose. In contrast to the polyacrylamide white water system, which has an anionic charge and has chemical attraction for a weak to strong cationic tree-formaldehyde resin, hydroxyethyl cellulose is a cationic viscosity modifier. While not wishing to be bound to a particular theory, it is believed that the addition of an anionic phosphate ester to the tree-formaldehyde resin acts to negate the cationic charge of hydroxyethyl cellulose that comes in contact with the resin on the glass fibers.

containing 22 to 25% solids was applied on the fiber mat and excess binder removed by vacuum. The mat was then placed in a Werner Mathis oven for 60 seconds at 205° C. to cure the resin.

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EXAMPLE 2

A commercially available urea-formaldehyde resin (GP 2928) was used as a control resin. This control resin, GP 2928 resin fortified with 23% polyvinyl actuate (PVAc), and resin modified with 0.5% ZELEC UNO (GP 328T67) were used as binder to prepare glass fiber mats as described in Example 1.

Seven 3"x5" cut samples were tested for tensile strength under dry conditions and after soaking in an 85° C. water bath for 10 minutes on an Instron with a crosshead speed of 2 inches and a jaw span of 3 inches. Tear strength was tested on 2.5"x3.0" cut samples using an Elmendorf Tear Machine. The mean values of all tests are shown in Table I.

TABLE I

Resins	Mat. WL*	% LOI	Dry Tensile ^b	Hot Wet Tensile ^b	% R	Tear
GP 1918 GP 1928 + 23% PVAc	1.80 1.75	24 22	117 115	81 75	59 65	390 380
GP 328T67 (+ 0.5% ZELEC UN @)	1.75	21	129	78	GO	515

"pounds per hundred square feer "pounds for a 3" wide sheet

Following application of the binder, the glass fiber mat is dewatered under vacuum to remove excess binder solution. The mat then is dried and incorporated binder composition is cured in an oven at clevated temperatures, generally at a temperature of at least about 200° C., for a time sufficient to cure the resin. The amount of time needed to cure the resin is readily determinable by the skilled practitioner. Heat treatment alone is sufficient to effect curing. Alternatively, but less desirably, catalytic curing in the absence of heat may be used, such as is accomplished with an acid catalyst, e.g., ammonium chloride or p-toluene sulfonic acid.

The finished glass mar product generally contains between about 60% and 90% by weight glass fibers and between about 10% and 40% by weight of binder, 15-30% of binder being most preferable.

The following examples are intended to be illustrative only and do not limit the scope of the claimed invention. 55

EXAMPLE 1

Glass fiber mats were prepared by adding 0.5 gms of surfactant (Katapol VP-532), 0.1 gms of defoamer (Nelco 60 2343) and 6.5 gms of Manville 1" cut glass fibers obtained from Schuller International to 7.5 liters of hydroxyethyl cellulose-containing white water having a viscosity of 12 to 14 cps and mixed for 3 minutes. Excess water was drained and then vacuum dewatered on a foraminated surface to 65 form a wet glass fiber mat. A urea-formaldehyde binder

Dry tensile strength, hot water tensile strength and precent retention (%R) of dry tensile strength under hot wet condition (hot wet/dry) of the urea-formaldehyde resin containing ZELEC UN® compare favorably to those of the control (urea-formaldehyde resin) and the latex fortified urea-formaldehyde resins. In contrast, the ZELEC UN® modified urea-formaldehyde resin produced a glass fiber mat having superior tear strength compared to the control urea-formaldehyde resin and the latex fortified urea-formaldehyde resin.

EXAMPLE 3 (COMPARISON)

Glass fiber mats were prepared as described in Example 1 except the hydroxyethyl cellulose white water system was replaced by a polyacrylamide white water system containing 0.02 to 0.1% polyacrylamide and having a viscosity of 4-10 cps, preferably 6 cps. A commercially available latex fortified urea formaldehyde resin (GP 2928 containing 23% PVAc), a commercially available urea-formaldehyde resin modified with a polyamine (GP 2942) and a urea formaldehyde resin containing 0.5% ZELEC UN® (GP 322T67) were used to cure the glass fiber mats as described in Example 2. Dry and hot wet tensile strength and tear strength was determined as described in Example 2. The results are show in Table II. The values shown in Table II are the ranges of the means of 5 studies, 7 samples per study.

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TABLE II

Rosins	Mat. Wt.	% LO1	Dry Tensile	Hot Wei Tensile	% R	Tear
GP 2928 23% PVAc	1.60-1.90	18-25	120-140	65-104	50-80	300-350
GP 2942 (+ polyamine modifier)	1.60~1,90	18-25	120-140	65–104	50-80	400~500
GP 328767 (+0.5% ZELEC UN ®)	1.60-1.90	18-25	120-140	65-104	50~80	300-350

EXAMPLE 4

Glass fiber mats prepared as described in the hydroxy. 15 Example 2. The mean values are shown in Table IV. ethyl cellulose white water system of Example 1 were cuted with the same resins used in Example 3 and tested for dry and hot wet tensile strength and tear strength as described in Example 2. The results (range mean values of 5 studies-7 samples per study) are shown in Table III.

and hot wet tensile strength and tear strength as described in

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TABLE III

Resins .	Mar Wi.	% LO)	Dry Tensile	Hot Wet Tensile	% R	Tear
GP 2928 + 23% PVAC	1.60-1.80	18-25	100-110	53-84	50-80	360-400
GF 2942 (+ polyamine modifier)	1.60-1.80	1825	1!0-120	39 -92	50-80	380-450
GP 328767 (+ 0.5% ZELEC UN @)	1.60-1.80	19-25	120-130	63–100	50-90	500-600

The use of a phosphate ester modified-resin provided 35 higher tear strength to glass mats prepared using a hydroxyethyl cellulose white water system. The high tear strength obtained in Examples 2 and 4 for glass mats prepared using

TABLE IV

Resins	Dry Tensile	Hat Wet Tensile	% Reservious	Tear Strength	Mat Wc.	% LQI
GP 2928 + 25% PVAc	139	96	70	350	1,50	29
GP 328T67 (+ ZELEC UN @)	140	89	. 63	490	08.1	28
GP 2928 (+ ZELEC TY ⊗)	141	104	74	300	1.90	23

the hydroxyethyl cellulose white water system could not be obtained using the polyacrylamide white water system of Example 3.

EXAMPLE 5

Glass fiber mats prepared as described in the hydroxyethyl cellulose white water system of Example 1 were cured with a commercially available latex fortified urea-formal- 60 dehyde resin (GP 2928 containing 25% PVAc), a ureaformaldehyde resin containing 0.5% ZELEC UNB (GP 328T67) or a urca-formaldehyde resin containing 0.5% ZELEC TY®. ZELEC TY® is a neutralized, water-soluble 65 amonic phosphate ester with a lower molecular weight fatty alcohol backbone. The glass fiber mais were tested for dry

As can be seen in Examples 2 and 4, resins modified with water-insoluble anionic phosphate esters, such as ZELEC UN®, provide significantly higher tear strength in glass mat 55 than latex fortified urea-formaldehyde resins when the glass mat is formed using a hydroxyethyl cellulose white water system. Although use of the water-soluble ZELEC TY® modified hinder gave dry and hot wet tensite strength equal to the latex fortified binder, the ZELEC TY® modified binder did not improve the tear strength properties compared to the latex fortified binder, as did the water-insoluble ZELEC UN® modified binder.

I claim:

1. A method of making a glass fiber mat comprising: dispersing glass fibers in an aqueous medium containing hydroxyethyl cellulose to form a slurry,

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passing the slurry through a mat forming screen to form a wet glass fiber mat,

applying a binder comprising a urea-formaldehyde resin and a water-insoluble, unneutralized anionic phosphate ester, a C₈ to C₁₆ fatty alcohol to said wet glass fiber 5 mat, and

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curing the binder.

2. The method of claim 1 wherein the anionic phosphate ester is present in an amount of from about 0.1% to about . 5.0% based on the weight of the binder.

RELATED PROCEEDINGS APPENDIX

NONE